

# Investigation of Intumescent Fabric Coatings For Protection Against Thermal Radiation and Flame

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## ABSTRACT

Evaluation of intumescent and miscellaneous organic fabric coatings for protection of personnel indicates that varying amounts of energy attenuation are provided and, therefore, some degree of protection is possible.

A new area of exploration has been opened with the current investigation of the use of intumescent materials on clothing as a possible means of protecting personnel against flame and thermal radiation. The National Aeronautics and Space Administration recently held a meeting at Moffet Field, Calif. (1), where some new intumescent paints containing aromatic nitroamine bisulfates were described. These materials were pointed out to be very effective in protecting various inanimate substrates, including canvas, against jet fuel fires and simulated solar radiation. More recently Parker et al. discussed the properties of p-nitroaniline bisulfate (2). However, no information is available on the application of these intumescent materials to clothing for protection of personnel against thermal radiation and flame. Similarly, very little information is available in the literature on any other intumescent materials applied to clothing. A few foam-forming materials were evaluated in 1960 by A. D. Little, Inc. (3), as additives to 5- and 9-oz cotton poplin. These additives, consisting generally of a polymer and blowing agent or a pre-foamed polymer, did not appreciably change the heat transfer properties of the poplin.

The idea of using intumescent materials on clothing as a means of protecting personnel against flame and thermal radiation is attractive for several reasons: (1) A large amount of protective char is generated by some intumescent formulations during exposure to heat; (2) the protec-

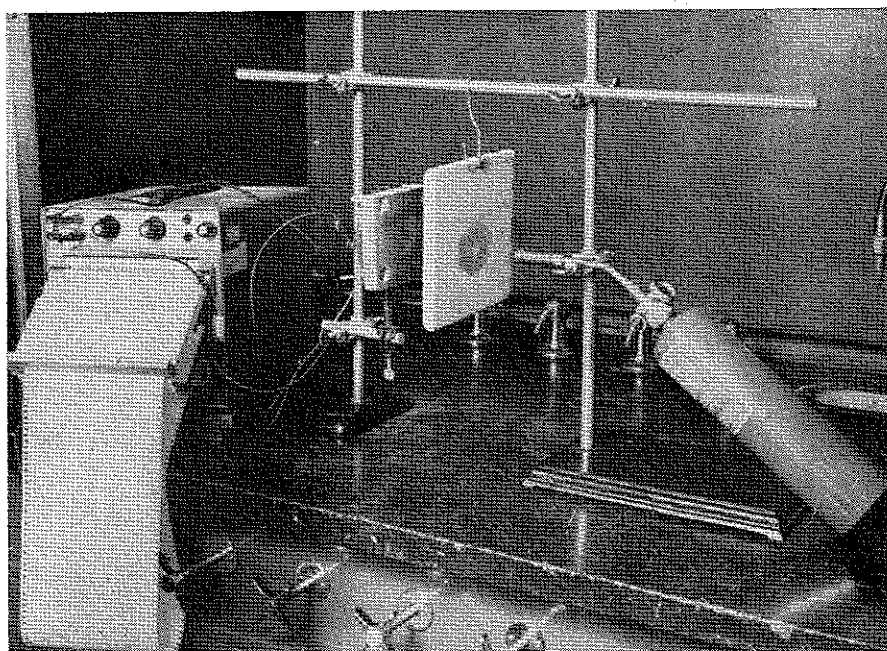


Figure 1  
Flame Test Apparatus

tive char is developed only when needed; (3) the protection capability is present at all times; and (4) the rate of heat transfer through the coatings and char appears relatively low.

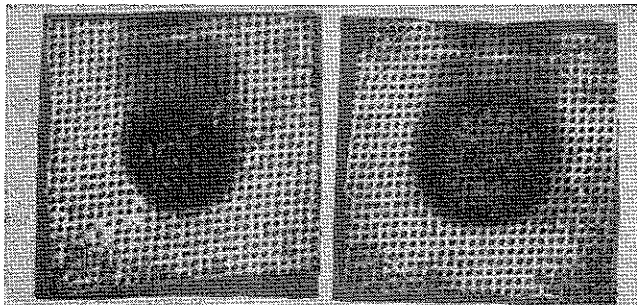
However, there are some potential disadvantages to intumescent coatings on fabrics when these materials are considered for personnel protection: (1) The coatings can appreci-

ably increase the weight of a fabric substrate and thereby the load carried by a person; (2) the air permeability of fabrics can be reduced appreciably (except when coatings are applied to mesh materials), thereby reducing the comfort of the individual; and (3) the coatings can require several seconds of heating to form large volumes of char during which

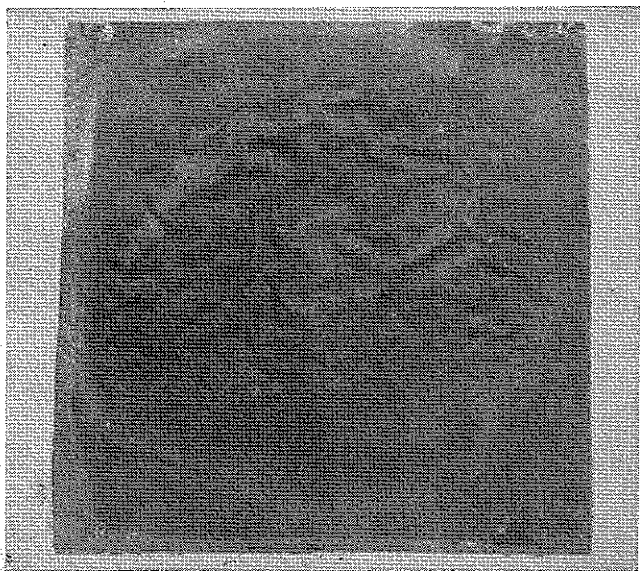
TABLE I  
Coatings on Non-Fabric Materials

| Formula                              | Material       | Coating Thickness (mils)* |               |        |
|--------------------------------------|----------------|---------------------------|---------------|--------|
|                                      |                | Brush Method              | Poured Method |        |
|                                      |                |                           | Average       | Range  |
| I (o-nitroaniline in nitrocellulose) | Aluminum sheet | 5                         | 70            | 40-100 |
|                                      | Asbestos board | 5                         | 70            | 40-100 |
| II (o-nitroaniline in polyurethane)  | Aluminum sheet | 15-20                     | 60            | 50-70  |
|                                      | Asbestos board | 15-20                     | 60            | 50-70  |

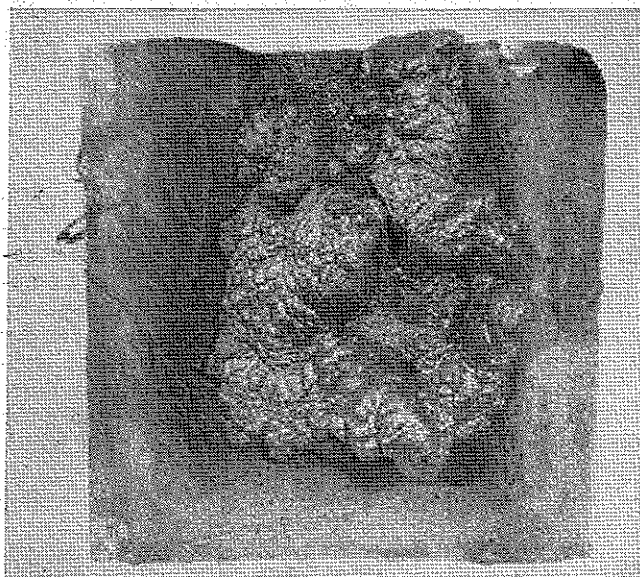
Air dried



Nylon mesh with Coating II, over uncoated fiber N-70. Carbon arc exposure: left, 20 cal; right, 10.3 cal



Unexposed Formula I on cotton duck

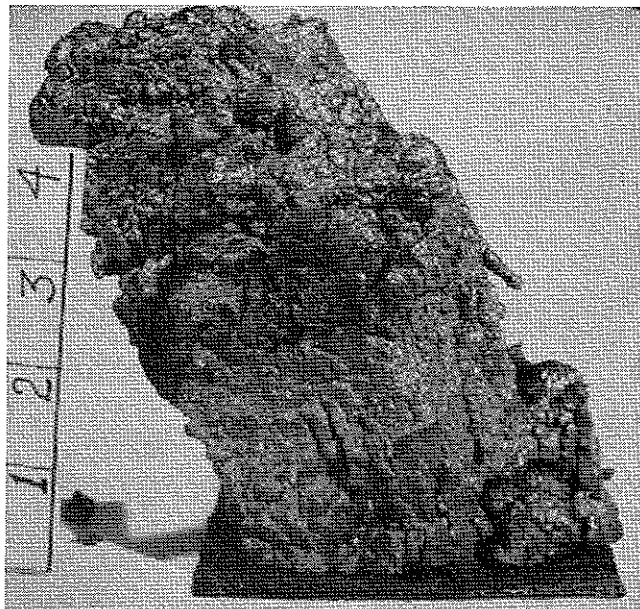


time the temperature behind the sample may rise high enough to cause a burn.

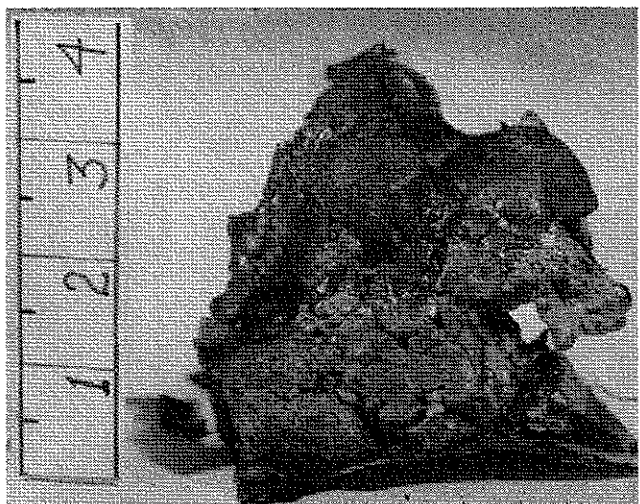
In spite of these potential disadvantages it seemed worthwhile to evaluate selected coatings and fabric combinations, especially since to our knowledge intumescent coatings had never been evaluated on heat resistant fabrics made with Nomex\* nylon fiber and the experimental Fiber N-

70. Some non-fabric substrates were also used for comparison. The evaluations were carried out with the aid of both a simple flame test apparatus designed and assembled in this laboratory and the U. S. Army Natick Laboratories (NLABS) arc-image furnace.

\*E. I. du Pont de Nemours & Co., Inc., trade name for a fire-resistant nylon fiber.



Cotton duck, 21.4 oz/sq yd, Formula I, 89 mil. Propane flame exposure: 10 sec;  $\Delta T$ , 1°C.



Cotton/ rayon cloth, Formula I, 26 mil. Propane flame exposure: 5 sec;  $\Delta T$ , 28°C.

Figure 2  
Intumescence Exhibited by Selected Materials  
Exposed to Carbon Arc and Propane Flame

Cotton/ rayon cloth, Formula I, 231 percent add-on. Propane flame exposure: 5 sec;  $\Delta T$ , 63°C.

## EXPERIMENTAL

The heat-resistant fabrics and other materials selected for evaluation were:

1. Wool: blanket, 13.8 oz/sq yd, 35 percent reprocessed; and serge 9.6 oz/sq yd.
2. Fiber N-70: poplin, 4.2 oz/sq yd.
3. Nomex: herringbone twill, 3.3 oz/sq yd.

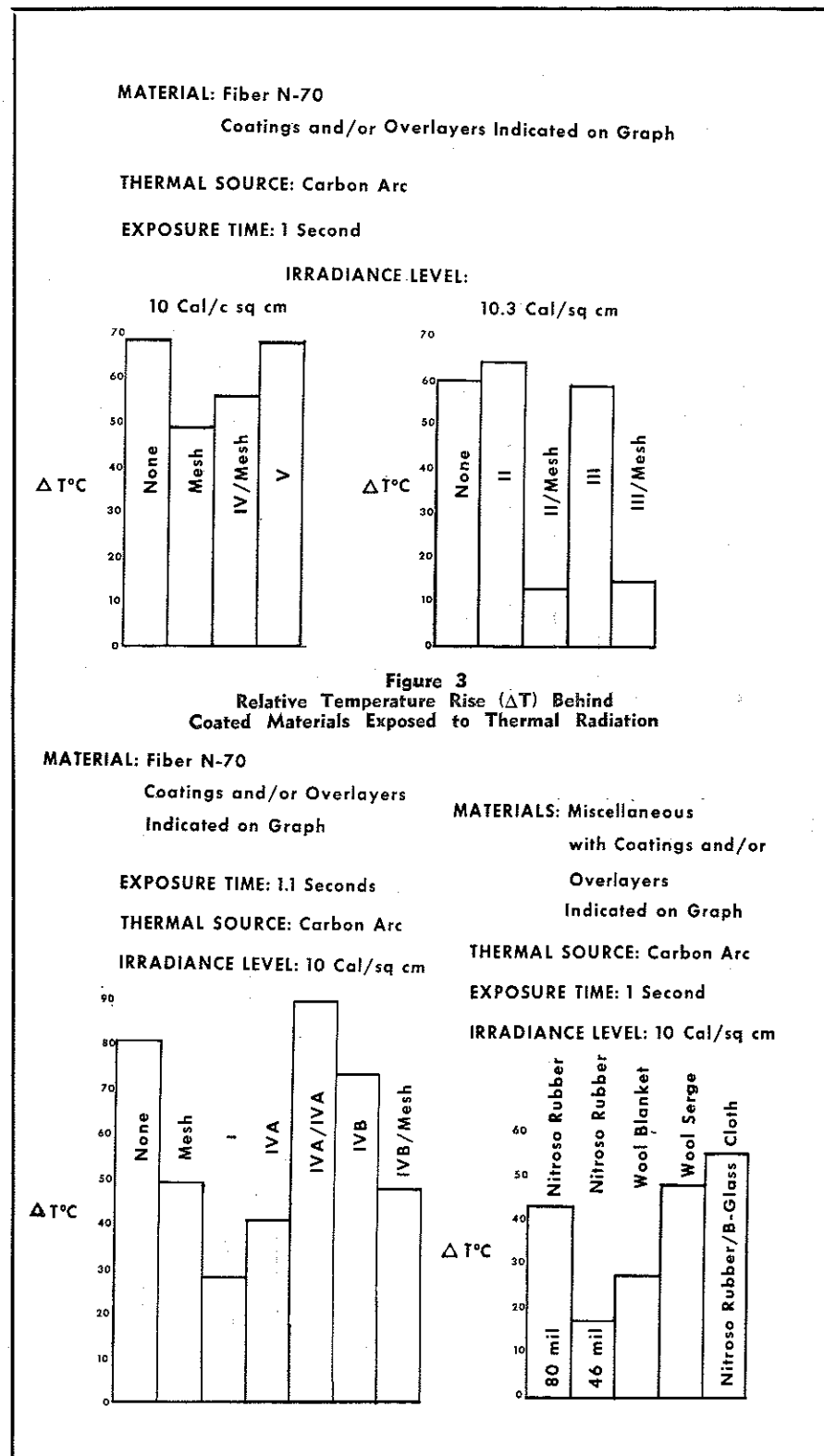
4. Nylon 66: 9 oz/sq yd mesh, Type MIL-C-8061.
5. Beta-glass cloth, factory-coated with carboxy nitroso rubber: cloth, 27 $\frac{3}{4}$  oz/sq yd; coating, 2 $\frac{1}{4}$  oz/sq yd of cloth.
6. Aluminum: 5 mil, polished sheet.
7. Asbestos:  $\frac{1}{2}$  in. asbestos board, unpainted.
8. Nitroso rubber: mesh 48 oz/sq yd and 102 oz/sq yd.
9. Cloth, cotton warp, rayon fill, 7.6 oz/sq yd.
10. Cotton duck, 21.4 oz/sq yd.
11. Cotton duck with FWWMR treatment, base fabric 9.85 oz/sq yd, coated weight 13.9 oz/sq yd.

All of these materials were Military Olive Green (OG) in color except for the coated  $\beta$ -glass, which was white. All but the nitroso rubber mesh materials and the experimental fiber N-70 fabric were available commercially. The nitroso rubber for the mesh was compounded with commercial fillers, such as oxides of iron, chromium and zinc, to achieve colors approaching that of the Military OG. The rubber was cured according to conventional curing processes in the form of 46- and 80-mil tensile sheets. These sheets were perforated with a cork borer (size number one) at the rate of 16 holes per sq in. to obtain the mesh.

The potentially intumescent coatings consisted of o-nitroaniline, sulfuric acid, and various binders, namely, nitrocellulose, polyurethane rubber, polyvinyl alcohol, and zein. In addition, char and/or smoke-forming coatings of the protein zein and a corn dextrin were prepared. Since o-nitroaniline in any form is especially toxic to humans according to Sax (4), this compound and the coatings containing it were handled with gloves in a well-ventilated area.

To make the coatings the following procedures were undertaken:

**Coating I** — o-Nitroaniline (27.5g) was added to a mechanically stirred solution consisting of nitrocellulose (20.7g commercial nitrocellulose cement, specifically, Ambroid Liquid Cement, 29 percent solids, Ambroid Co., Inc., Weymouth, Mass.) and methyl ethyl ketone (MEK) (19g, 23.8 ml) until all particles had dissolved. Sulfuric acid (19.7g, 11.5 ml) was dissolved in 13.1g (16.4 ml) absolute ethanol and then added dropwise to the nitrocellulose solution as the stirring continued. During the addition, the bisulfate salt of o-nitroaniline precipitated and heat was liberated. Vigorous stirring continued for two hours, with occasional pauses to determine if the salt would remain in suspension. Since it did not



the solution was constantly stirred during the coating process.

**Coating II** — Polyurethane rubber cement, 46.3g (Bostic® adhesive, 15 percent solids, USM Corp., Cambridge, Mass.), was diluted with 6.5g (7.3 ml) of tetrahydrofuran (THF). To this solution was added 27.5g of o-nitroaniline with mechanical stirring until all particles dissolved. This was followed by dropwise addition of 19.7g (11.5 ml) of concentrated sul-

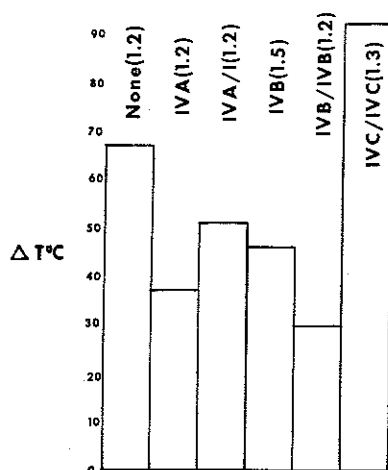
furic acid with continued stirring. As in Coating I, the bisulfate salt precipitated and constant stirring was needed to keep it in suspension.

**Coating III** — o-Nitroaniline, 27.5g, was mixed by mechanical stirring with 287.1g of a polyvinyl alcohol solution (Partial Film No. 10, 7 percent Solids, Boatex Fiberglass Co., Inc., Needham, Mass.) after which 19.7g (11.5 ml) of concentrated sulfuric acid was added with continued

MATERIAL: Fiber N-70

Coatings and/or Overlayers and Exposure Times(Seconds) Indicated Below on Graph

THERMAL SOURCE: Propane Torch



MATERIAL: Nomex

Coatings and/or Overlayers and Exposure Times(Seconds) Indicated Below

THERMAL SOURCE: Propane Torch

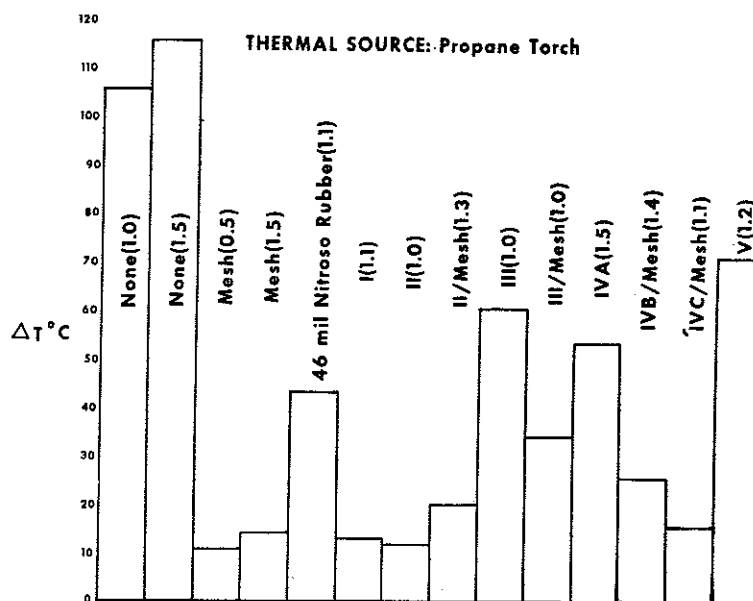
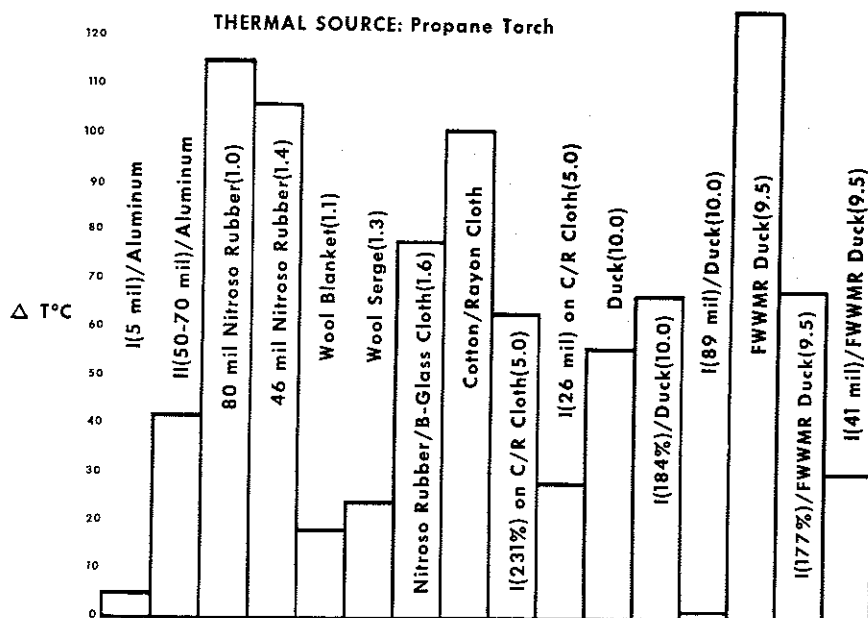


Figure 3 (cont.)  
Relative Temperature Rise (ΔT) Behind Coated Materials Exposed to Thermal Radiation

MATERIALS: Miscellaneous

with Coatings and/or Overlayers and Exposure Times(Seconds) Indicated on Graph

THERMAL SOURCE: Propane Torch



stirring. During each of these steps only part of the o-nitroaniline dissolved. It was completely dissolved by heating the mixture to 89°C on a steam bath with constant stirring. The solution was then cooled, whereupon a very fine precipitate appeared. This precipitate remained suspended for more than 24 hours. No additional solvent was used.

**Coating IVA** — Zein, 10g, (Corn Products Sales Co.) was dissolved

with occasional swirling in a 70 percent by volume isopropyl alcohol solution. The alcohol solution was prepared by mixing 70cc (54.6g) of the alcohol with 30 ml (30g) of water. When the zein was completely dissolved, 2.74 ml more water was added. The resulting solution was slightly viscous.

**Coating IVB** — This solution was prepared similarly to coating formula IVA except that 1.37 ml of 38 percent

formaldehyde solution was substituted for the extra water. The weight of formaldehyde was 0.5g, that is, 5 percent based on the weight of zein.

**Coating IVC** — A zein solution was prepared according to coating formula IVA to which was added 9.2g of o-nitroaniline. The mixture was heated under hot tap water to help dissolve the o-nitroaniline. It was then cooled and 3.8 ml of concentrated sulfuric acid was added dropwise with stir-

ring. All solids dissolved completely.

**Coating V** — Dextrin, 20g, (Hercules, Inc.) was added in portions with stirring to 80 ml of water until all particles were dissolved.

### Test Sample Preparation

The fabric and non-fabric materials were prepared for testing by cutting them into approximately 4 x 4 in. specimens and coating them directly with the mixtures and solutions described without a "sealer" or finish coat. The coatings were applied to the materials with a paint brush except in a few instances where they were poured on the materials. These coating methods were adequate for preliminary evaluations but they did not produce coatings of uniform thickness, partly because there were relatively large particles of solid suspended in many of the coating mixtures. The safety precautions used in handling the o-nitroaniline-containing coatings were also used in handling the respective coated materials.

The coated test samples are described in Tables I and II. Uncoated control samples of each material were also prepared. The wool blanket, wool serge, and nitroso rubber were tested in an uncoated state only.

The NLABS arc-image furnace, which was used for the irradiation screening tests, was described previously by McCue (5). The flame test apparatus constructed in this laboratory consisted of a sample holder, propane torch, shutter, and recording potentiometer. These components were assembled as indicated in Figure 1.

| Coating                                   | Material     | Method of Drying | Number of Coats | Add-on by weight (%) |
|---|--------------|------------------|-----------------|----------------------|
| I (o-nitroaniline in nitrocellulose)      | Nylon mesh   | air              | 1               | 119.0                |
| II (o-nitroaniline in polyurethane)       | Nomex        | air              | 1               | 239.0                |
|   | Fiber N-70   | "                | 1               | 128.0                |
|   | Nylon mesh*  | "                | 1               | 121.0                |
|   | Nylon mesh** | "                | 1               | 112.0                |
| III (o-nitroaniline in polyvinyl alcohol) | Nomex        | air              | 1               | 126.0                |
|   | Fiber N-70   | "                | 1               | 74.0                 |
|   | Nylon mesh*  | "                | 1               | 48.5                 |
|   | Nylon mesh** | "                | 1               | 45.0                 |
| IVA (zein)                                | Fiber N-70   | air              | 1               | 13.6                 |
|   | Fiber N-70   | oven             | 1               | 10.2                 |
|   | Fiber N-70   | oven             | 2               | 25.2                 |
|   | Nylon mesh   | oven             | 1               | 8.1                  |
| IVB (zein with formaldehyde)              | Fiber N-70   | air              | 1               | 15.0                 |
|   | Fiber N-70   | oven             | 1               | 14.3                 |
|   | Fiber N-70   | oven             | 2               | 32.0                 |
| IVC (zein with o-nitroaniline)            | Fiber N-70   | air              | 1               | 26.5                 |
|   | Fiber N-70   | "                | 2               | 48.3                 |
|   | Nylon mesh   | "                | 1               | 20.7                 |
| V (dextrin)                               | Fiber N-70   | air              | 1               | 44.2                 |
|   | Nomex        | "                | 1               | 77.0                 |
|   | Nylon mesh   | "                | 1               | 24.8                 |
| Nitroso rubber                            | 8-glass      | —                | —               | 7.5                  |

\*Used with untreated Nomex

\*\*Used with untreated Fiber N-70

The sample holder consisted of a transite block containing a copper-constantan thermocouple embedded in its front surface. The holder and the propane torch were positioned so that the tip of the torch was 5 in. from the front surface of the holder. The shutter, suspended between the holder and the torch, was a piece of

1/2-in. asbestos board. The torch was a Turner LP-5 propane torch with an LP-603 pencil point burner. When exposing a sample, the temperature rise ( $\Delta T$ ) behind the sample was registered in millivolts on a Honeywell Electronik 19 recorder which was connected to the thermocouple in the sample holder.

TABLE III  
Behavior of Materials Irradiated in the Carbon Arc Image Furnace

| Material                          | Coating formula                     | Add on (%) | Flame during exposure | Smoke formation  | Appearance after exposure                                     | Temperature rise behind material ( $\Delta T$ °C) |
|-----------------------------------|-------------------------------------|------------|-----------------------|------------------|---|---|
| Nylon Mesh/<br>Fiber N-70         | no coating                          | no add-on  | No                    | None             | mesh fibers fused;<br>some char at high $\Delta T$            | 23.7<br>to<br>54.8                                |
| Wool<br>Blanket                   | no coating                          | no add-on  | No                    | much white smoke | some char; back side<br>intact                                | 28.3  |
| Nitroso<br>rubber<br>mesh, 48 oz. | no coating                          | no add-on  | No                    | much smoke       | surface etched and<br>covered with friable<br>black particles | 16.9<br>to<br>19.5                                |
| 8-glass                           | carboxy-<br>nitroso rubber<br>(CNR) | 7.5        | No                    | very little      | no change   | 54.8  |
| Nylon Mesh/<br>Fiber N-70         | Formula I/<br>no coating            | 119/0      | Yes                   | yellow smoke     | some intumesced char<br>on mesh                               | 28.3  |
| Nylon Mesh/<br>Fiber N-70         | Formula II/<br>no coating           | 112/0      | Yes                   | yellow smoke     | some intumesced char<br>on mesh                               | 13.2  |
| Nylon Mesh/<br>Fiber N-70         | Formula III/<br>no coating          | 45/0       | Yes                   | yellow smoke     | mesh charred/ Fiber N-70<br>slightly charred                  | 16.7  |

## Test Procedures

Temperature rise ( $\Delta T$ ) determined in the NLABS are-image furnace. A sample was attached with two rubber bands to the sample holder so that there was as much contact as possible between the sample and the thermocouple in the holder. Each sample, consisting of either a single cloth swatch or a mesh swatch over a cloth swatch, was exposed at approximately 10 cal/cm<sup>2</sup> for 1 sec. The

appearance of each sample before and after exposure and the behavior of each sample during exposure were observed. Also, the temperature rise at the back surface of the sample was recorded in millivolts. During the exposures, dark glasses were worn by personnel to protect their eyes from the radiation while observing the samples.

Temperature rise ( $\Delta T$ ) determined in a propane torch flame. A sample was attached as described above to

the sample holder and the recorder was adjusted to the appropriate speed and attenuation settings. The torch was then ignited and the valve opened wide. Each sample was then exposed to the flame on a reverse countdown of three. At 3, all was in readiness; at 2, the recorder was turned on; and at 1, the shutter was removed and a stop watch was started simultaneously. At zero, the shutter was closed and the watch was stopped. As in the above procedure, the appear-

TABLE IV  
Behavior of Materials Exposed to a Propane Torch Flame

| Material             | Coating formula            | Add-on                   | Exposure time (sec) | Combustion during exposure            | Smoke formation                         | Appearance after exposure   | Temperature rise behind material ( $\Delta T$ °C) |
|----------------------|----------------------------|--------------------------|---------------------|---------------------------------------|---|---|---|
| Nomex                | no coating                 | no add-on                | 1-1.5               | yes, almost none at lowest $\Delta T$ | none                                    | large hole; some melting; damage small at lowest $\Delta T$   | 18 to 115.5                                       |
| Nylon mesh/<br>Nomex | no coating                 | no add-on                | 0.5-1.5             | none                                  | none                                    | mesh melted; holes in Nomex at higher $\Delta T$  | 10.5 to 35, possibly greater                      |
| Wool blanket         | no coating                 | no add-on                | 1.1                 | yes                                   | none                                    | nap burned off; some char; back surface unchanged   | 13  |
| Wool serge           | no coating                 | no add-on                | 1.3                 | none                                  | none                                    | some char; back surface unchanged   | 24  |
| Aluminum             | I                          | 5 mil coating            | 1-7                 | none                                  | yellow smoke                            | intumesced char $\frac{3}{8}$ " to $\frac{1}{4}$ " high   | 0-5*  |
| Asbestos             | I                          | about 5 mil coating      | 10                  | none                                  | none except when flame stopped          | intumesced char $\frac{1}{8}$ " high  | not recorded                                      |
| Aluminum             | I                          | 70 mil coating           | 15                  | none                                  | yellow smoke                            | intumesced char 5-6" high   | not recorded                                      |
| Asbestos             | I                          | 50-100 mil coating       | 17                  | none                                  | a little; some yellow and white smoke   | intumesced char $1\frac{1}{4}$ " high   | not recorded                                      |
| Nylon mesh/<br>Nomex | Formula I/<br>no coating   | 119%/0%                  | 1-5                 | none                                  | yellow smoke                            | some intumesced char at low $\Delta T$ , many mesh holes plugged; holes in Nomex at high $\Delta T$ | 11.5 to 14.0                                      |
| Asbestos             | II                         | about 5 mil coating      | 10                  | none                                  | puff of yellow smoke when flame stopped | intumesced char $\frac{1}{4}$ " high  | not recorded                                      |
| Aluminum             | II                         | 50-70 mil uneven coating | 30+                 | none                                  | yellow smoke                            | intumesced char 9/16" high; hole in aluminum  | 42*   |
| Asbestos             | II                         | 50-70 mil coating        | 24                  | none                                  | puff of yellow smoke when flame stopped | intumesced char $\frac{3}{4}$ " high  | not recorded                                      |
| Nomex                | II                         | 239%                     | 1                   | none                                  | yellow smoke                            | some intumesced char about $\frac{1}{4}$ " high   | 10.5 to 13  |
| Nylon mesh/<br>Nomex | Formula II/<br>no coating  | 121%                     | 1-1.5               | sometimes                             | yellow smoke                            | intumesced char; at higher $\Delta T$ some mesh holes filled with char and some holes in Nomex      | 14 to 26.5  |
| Nylon Mesh/<br>Nomex | Formula III/<br>no coating | 48.5%                    | 1                   | none                                  | yellow smoke                            | char and melting; holes in Nomex  | 29-37.5   |

\*Thermocouple not in good contact with aluminum sheet.

ance of a sample before and after exposure and its behavior during exposure were noted. Also, the temperature rise as indicated by the thermocouple was determined. Most of the exposures lasted 1-2 sec, but a few samples were exposed up to 30 sec. This procedure was rapid and provided reasonably consistent exposure times for easy comparison of results.

## RESULTS AND DISCUSSION

Intumescence was observed in varying amounts only on the materials having Coatings I and II containing the nitrocellulose and polyurethane binders, respectively. Examples of this intumescence are shown in Figure 2. The other materials produced merely smoke or vapors and/or unfoamed char. These types of behavior occurred in both the irradiance and the flame tests. Since Goodwin et al. (1) reported intumescent action in nitroamine bisulfates alone, the above evidence indicates that some binders can interfere with this phenomenon as recognized by Parker et al (2). Thus the binder could play an important role in the intumescent reaction.

The amount of intumescence observed during the tests increased with the exposure, that is, with the irradiance level or the number of cal/cm<sup>2</sup>/sec in the irradiance test and

with the exposure time in the flame test. This increase was noticed, for example, with the coated nylon mesh which, it was hoped, would show enough intumescence to fill the mesh holes. However, most of the char foamed perpendicular to the plane of the samples toward the energy source rather than laterally in the plane of the sample, and thus most of the holes did not fill in. The greatest amount of intumescence was observed with a very thick coating of nitrocellulose formulation on aluminum sheet. When exposed to the flame for about 15 sec this sample increased in thickness from 70 mils to 5 or 6 in., an 8,000 percent increase. This result agreed well with the results of Goodwin et al. (1) and served as a standard for comparison.

Several of the materials screened in the irradiance and flame tests showed a high degree of energy attenuation as indicated by the relatively low temperature rise ( $\Delta T$ ) behind the samples during exposure. These "low  $\Delta T$ " materials are shown in Tables III and IV with their  $\Delta T$  values and other observations of their behavior. The remaining materials exhibited  $\Delta T$  values from 30 up to 150°C. Examples are shown in Figure 3. It is interesting to note that several of the low  $\Delta T$  materials exhibited no intumescence and some were uncoated.

A satisfactory  $\Delta T$  value with re-

spect to personnel protection in these screening tests was arbitrarily selected as a maximum of 10 to 20°C, the lower temperature being preferable. Although this rise in temperature is insignificant for inanimate objects, it may or may not produce a burn on human skin. On this basis, the best materials in the irradiance test were nitroso rubber mesh by itself and the composites of nylon mesh with a polyurethane or polyvinyl alcohol coating over Fiber N-70. The best materials in the flame test were the wool blanket, Nomex, nylon mesh over Nomex, Nomex with the polyurethane coating, and the composites of nylon mesh with a nitrocellulose or polyurethane or zein-o-nitroaniline coating over Nomex. However, these materials did not necessarily exhibit  $\Delta T$  values consistently in the 10 to 20°C temperature range. Thus, only a limited amount of protection for personnel would be obtainable with these materials, but their properties are attractive for applications where personnel are not concerned. Table V summarizes the behavior and potential protection for personnel afforded by all the combinations of coatings and fabrics tested.

## ACKNOWLEDGEMENTS

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TABLE V  
Behavior and Protection Afforded by  
Coated Fabric Combinations and Miscellaneous  
Materials

| Materials  | Exposure source      | Behavior and protection                                |
|--|----------------------|--|
| 1. Nitroso rubber mesh   | carbon arc           | smoke formation, limited protection                    |
| 2. Nitroso rubber mesh over Nomex  | flame                | no protection  |
| 3. Nitroso rubber coated glass cloth   | carbon arc and flame | smoke sometimes observed, no protection                |
| 4. Nomex alone and nylon mesh over Nomex   | flame                | limited protection                                     |
| 5. Nomex with polyurethane (very high add on)  | flame                | smoke formation, limited protection                    |
| 6. Nylon mesh with nitrocellulose, polyurethane or zein-nitroaniline coatings over Nomex | flame                | char and smoke, limited protection                     |
| 7. Nylon mesh with polyurethane and polyvinyl alcohol coatings over Fiber N-70           | carbon arc           | char and smoke, limited protection                     |
| 8. Wool blanket  | carbon arc and flame | smoke and char, no or limited protection, respectively |
| 9. Wool serge  | carbon arc and flame | smoke and char, no protection                          |
| 10. Dextrin on Fiber N-70 and Nomex  | carbon arc and flame | char, no protection                                    |
| 11. Zein or zein-formaldehyde on Fiber N-70 and Nomex                                    | carbon arc and flame | gases, no protection                                   |